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The objective of the program is to develop a fundamental understan ding of YBCO film deposition by Metal Organic Chemical Vapor Deposition 1'MOCVD,) on biaxially textured metal substrates. MOCVD was chosen for several reasons. First, other than PLD only MOCVIJ has been shown to achieve high deposition rates in producing high quality YBCO as shown in Table I. Second, only MOCVD offers the advantage of both high deposition rate and large deposition area, which is important for high throughput. This is shown in Table II. MOCVD offers other advantages too such as not being limited to line-of-sight deposition, separation of precursors from the deposition chamber (easy refill of precursors for unlimited regeneration), ability to modify film composition during deposition, and no target fabrication expense. When this program was begun 5 years ago, MOCVD of YBCO films suffered from problems such as reproducibility and stability Also, no group had demonstrated high Jo and high Ic on biaxially-textured metal substrates. Further, no group had demonstrated high ic and high Ic in YBCO films deposited in a moving mode. This program took upon the challenges to address to issues. The program includes a study of the a) influence of MOCVD processing conditions such as the flow rate of precursor vapors, precursor vaporization temperatures, oxygen partial pressure, reactor pressure, and the deposition temperature on the film features such as superconducting phase formation, composition, texture, deposition rates, uniformity in thickness, porosity and the presence of secondary phases, b) relationship between film microstructure and the critical current density (is), and c)influence of metal substrate and buffer layers on the growth and performance of YBCO d)development of MOCVD

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V. Selvamanickam, Program Manager

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Objectives :

The objective of the program is to develop a fundamental understanding of YBCO film deposition by Metal Organic Chemical Vapor Deposition (MOCVD) on biaxially-textured metal substrates. MOCVD was chosen for several reasons. First, other than PLD only MOCVD has been shown to achieve high deposition rates in producing high quality YBCO as shown in Table I. Second, only MOCVD offers the advantage of both high deposition rate and large deposition area, which is important for high throughput. This is shown in Table II. MOCVD offers other advantages too such as not being limited to line-of-sight deposition, separation of precursors from the deposition chamber (easy refill of precursors for unlimited regeneration), ability to modify film composition during deposition, and no target fabrication expense. When this program was begun 5 years ago, MOCVD of YBCO films suffered from problems such as reproducibility and stability. Also, no group had demonstrated high Jc and high Ic on biaxially-textured metal substrates. Further, no group had demonstrated high Jc and high Ic in YBCO films deposited in a moving mode. This program took upon the challenges to address to issues. The program includes a study of the

a) influence of MOCVD processing conditions such as the flow rate of precursor vapors, precursor vaporization temperatures, oxygen partial pressure, reactor pressure, and the deposition temperature on the film features such as superconducting phase formation, composition, texture, deposition rates, uniformity in thickness, porosity and the presence of secondary phases,
b) relationship between film microstructure and the critical current density (J_c), and
c) influence of metal substrate and buffer layers on the growth and performance of YBCO
d) development of MOCVD hardware specifically for fabrication of high-quality YBCO on metal substrates.

Table I Comparison of deposition rates achieved in various coated conductor processes

Process	*Deposition Rate for $Jc > 1\ MA/cm^2$ (Angstroms/second)*
PLD	650
MOCVD	150
E-beam BaF_2 (conversion)	1
MOD (conversion)	1

Table II Comparison of throughput in various coated conductor processes

Process	*Deposition Rate*	*Deposition Area*
PLD	High	Small
MOCVD	**High**	**Large**
E-beam BaF_2 (conversion)	Low	Large
MOD (conversion)	Low	Large

Status of Effort :

Over the course of this 5-year program, dramatic improvements have been made to the MOCVD process and hardware. Process conditions have been optimized to achieve reproducibly high performance and long-term stability. Hardware for precursor delivery, vaporization, and injection were all developed to achieve reproducibly high performance and long-term stability. Record-high Jc of 1.3 MA/cm^2 corresponding to a Ic of 130 A/cm has been achieved in short samples produced by MOCVD on metal substrates. Also, a continuously moving process was developed to demonstrate high Jc and Ic in longer samples – as high as 135 A over 6 cm. The excellent performance was achieved through optimizing the microstructure by developing optimum process conditions and MOCVD hardware.

Experimental :

MOCVD of YBCO has been conducted in custom-built facility at IGC-SuperPower. Over the course of the 5-year program, this facility has undergone numerous changes as will be described in this report. The schematic of our very first facility is shown in fig. 1. This system used solid-precursor delivery system where 3 individual precursors (for Y, Ba, and Cu) were separately sublimed in 3 ovens. The vaporized precursors were transported using a carrier gas such as Ar and mixed together in a mixing manifold. Oxygen was then added to this vapor mixture and then transported to the MOCVD reactor. Our first MOCVD reactor was of hot-wall type using a quartz chamber heated from outside using halogen lamps. No specific showerhead was used to inject the precursors. Figure 2 shows a photograph of our first MOCVD facility.

The metal substrates used in this work were nickel alloy based. A biaxially-textured oxide layer was deposited on this substrate using ion beam assisted deposition (IBAD). In addition to the IBAD substrates, single crystal yttira-stabilized-zirconia (YSZ) and $SrTiO_3$ substrates were also used for deposition to serve as a comparison. Tetramethyl heptanedionate (thd) precursors were used for Y, Ba, and Cu. YBCO films, 1 micron in thickness were deposited within a temperature regime of 700 to 800°C and in a reactor pressure of 1 to 5 Torr.

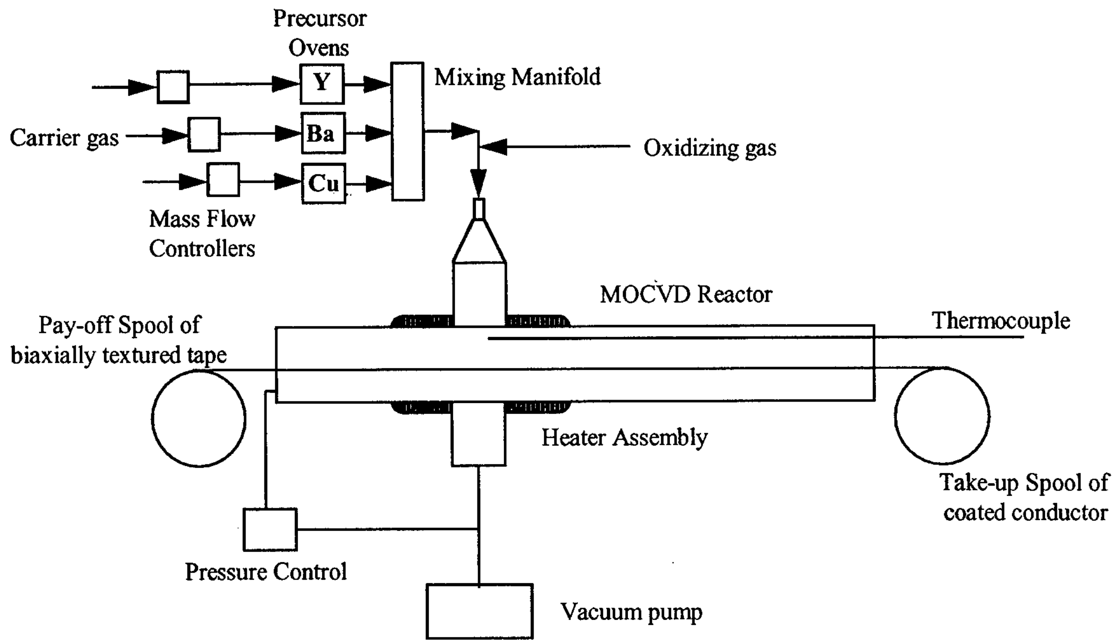


Fig. 1. Schematic of the first MOCVD facility established at IGC.

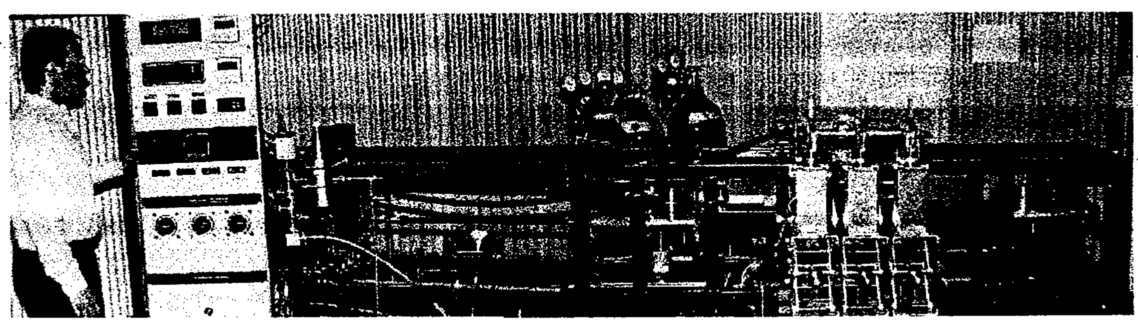


Fig. 2. Photograph of the first MOCVD facility established at IGC.

The out-of-plane and in-plane textures of YBCO films were examined at Intermagnetics by X-ray Diffraction including polefigure measurements. The surface morphology of the YBCO films was examined by Scanning Electron Microscopy followed by compositional analysis by Energy Dispersive X-ray Spectroscopy (EDS). The thickness of the films was measured by surface profilometry. The Jc of the films were measured using transport technique with a standard 4-probe method.

Accomplishments/New Findings :

The Materials Science Triangle approach, namely establishing correlation among process, microstructure, and properties was elaborately used throughout this program. The specific parameters that were examined within each category are listed below :

- Precursor Vaporization Temperatures
- Precursor Flow Rate
- Oxygen Partial Pressure
- Reactor Pressure
- Deposition Temperature
- Substrate & Buffer Properties

- YBCO Phase Formation
- Film Composition
- Texture
- Growth Morphology
- Deposition Rate
- Thickness Uniformity
- Secondary Phases
- Porosity

- Transition Temperature
- Critical Current Density
- Critical Current

Details of the correlation were provided in the progress reports over the last 4 years and will not be discussed again in this report except for the following example.

- Film growth rate ↑ as Precursor Flow Rate ↑
  Reactor Pressure ↓

  Particulate Formation ↑ as Precursor Flow Rate ↑
  Reactor Pressure ↑
- Previously,
  High Reactor Pressure (2.5 Torr) at low flow rates (~ 1 lpm) : Low growth rate, Particulate formation
- Using Modified hardware, found improved conditions :
  Low Reactor Pressure (1.6 Torr) at high flow rates (~ 5 lpm) : Higher growth rate, Reduce Particulates

Specific Issues with MOCVD :

In addition to issues that are common to all YBCO film processing techniques, there were issues that were specific to MOCVD that were addressed in this program. These issues are listed below.

- **Precursor Selection :**
  Thermal Stability, No interaction between precursors
  The vaporization temperatures of precursors should be close
  High vapor pressure
- **Vaporizer Design :**
  Temperature Uniformity
  Large Vaporization Area
- **Reactor Design :**
  Flow Dynamics & Heat Transfer to avoid convection-driven recirculation cells
- **Showerhead Design :**
  Avoid premature decomposition of precursors
  Uniform distribution of precursors over heated substrates

This study led to improvements in each of the following areas :

Improved Reactor Design
Improved MOCVD Precursor Delivery Scheme
Improved Precursor Vaporization Process
Improved Precursor Injection

Improved Reactor Design :

The hot-wall reactor used initially (shown in figs.1 and 2) led to several issues with reproducibility in YBCO performance. One of the main reasons was that the hot reactor wall affected the flow of the precursors. Precursors were found to be deposited on the reactor wall which affected the deposition rate on the substrate. Moreover, changing coating thickness affected the temperature stability during long deposition runs.

Therefore, we modified the reactor to a cold wall type. Instead of a quartz deposition chamber, a metal chamber was used. Heating was accomplished within the chamber. A photograph of the improved version of the MOCVD system with a cold-wall reactor is shown in fig. 3.

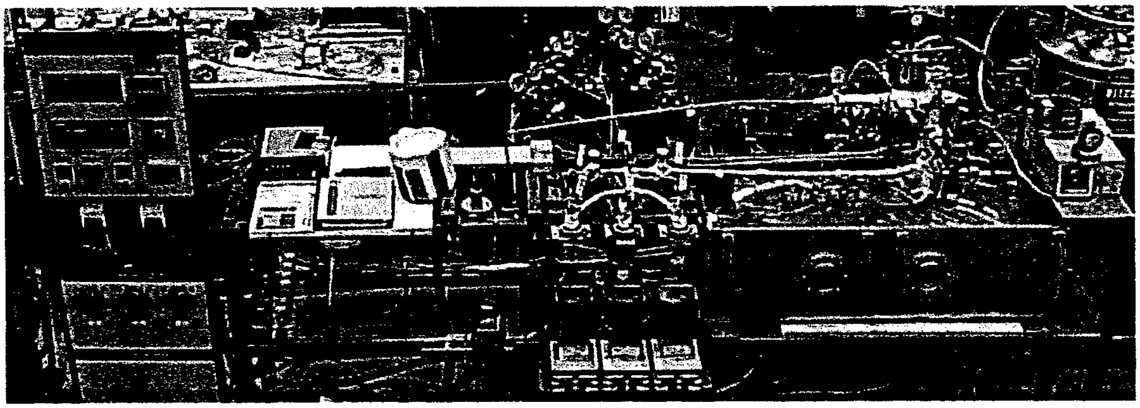


Fig. 3 Photograph of the modified MOCVD facility with a cold-wall reactor.

Improved MOCVD Precursor Delivery Scheme:

In the first 2 years of the program, a solid-precursor delivery scheme, described in fig. 1 was used. This led to numerous problems which are listed in Table III.

In order to avoid these problems, a liquid precursor delivery scheme was indigenously developed. The advantages of the liquid precursor delivery scheme are listed in Table 3. Fig 4 shows a schematic of the liquid precursor delivery scheme. Here, all 3 precursors are prepared in a solution form and mixed together. The mixed solution is pumped using a low-flow-rate delivery pump to a flash vaporizer. The solution is instantaneously vaporized and the vapor is then mixed with oxygen and delivered to the showerhead. A photograph of the MOCVD facility with this modification is shown in fig. 4.

Table III Comparison of solid-and liquid-precursor delivery schemes

	Solid-state Precursor	Liquid-state Precursor
Technique	Precursors are individually sublimed separately from 3 separate ovens	Precursor is flash vaporized at a single location
Temperature Control	Difficult (film composition is sensitive to even 1°C change)	Single-point of control. Easier
Precursor Stability	Bad for long deposition periods	More stable in solution form
Precursor Vapor Pressure	Changes as precursor is depleted	Constant volume of precursor is vaporized. Vapor pressure is constant
Deposition Rate	Difficult to increase due to turbulence of carrier gas at high rates	Easily increased by increasing precursor flow rate.

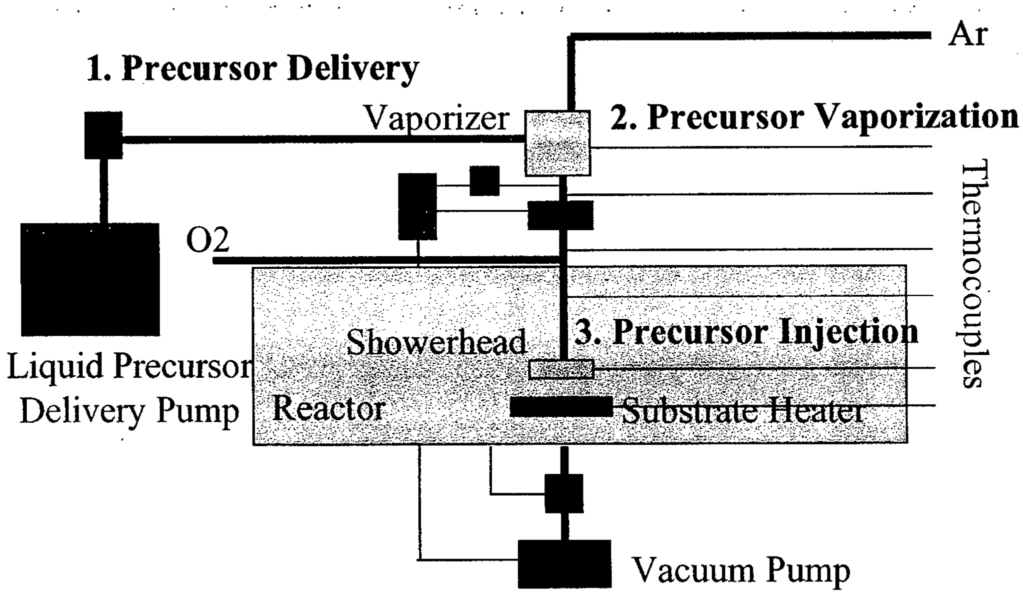


Fig. 3 Schematic of the MOCVD facility with a liquid precursor delivery system

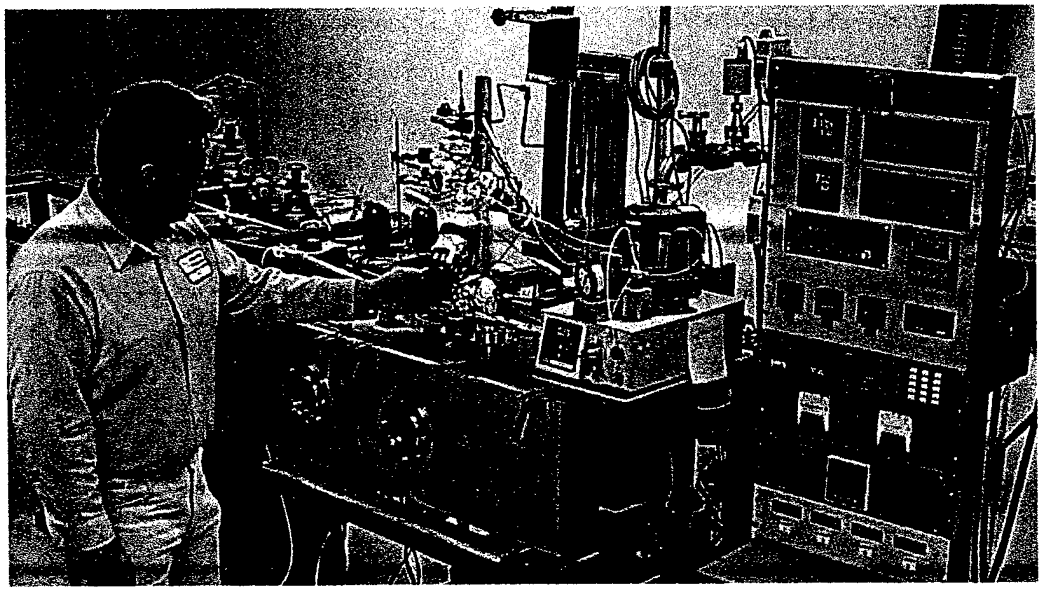


Fig. 4 Photograph of the MOCVD facility with a liquid precursor delivery system

Improvement to vaporizer :

Substantial improvements were made to the vaporizer, all of which are discussed in detail in our patent application which is now published as PCT WO 02/056420 A2. In essence, the vaporizer design avoided clogging, coating on the vaporizer walls, and premature precursor decomposition etc. A schematic of this vaporizer is shown in fig. 5.

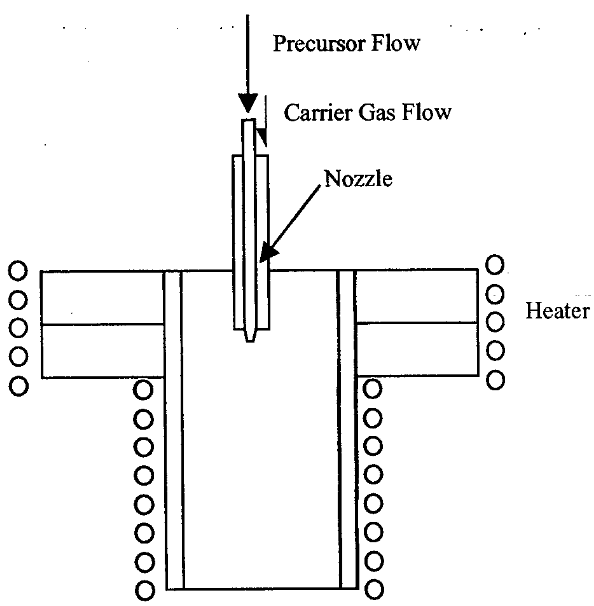


Fig. 5 Schematic of improved vaporizer

Improvement to showerhead :

Substantial improvements were made to the showerhead, all of which are discussed in detail in our patent application which is now published as PCT WO 02/056420 A2. In essence, the showerhead design resulted in uniform deposition and avoided premature decomposition of the precursors.. A schematic of this showerhead is shown in fig. 6.

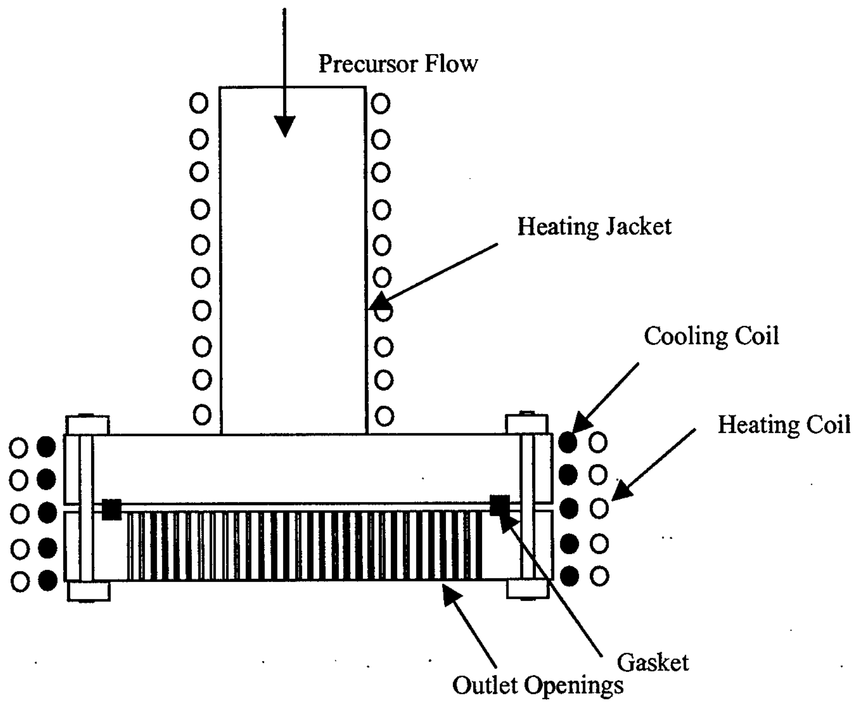


Fig. 6 Schematic of improved showerhead

Demonstration of record-high Jc on metal substrates :

All the above modifications, along with optimization of process parameters led to achievement of record-high Jc on metal substrates. Critical current density as high as 1.3 MA/cm^2 corresponding to a critical current of 130 A was achieved as shown in fig. 7. This high current density was maintained in presence of high magnetic fields too as shown in fig. 10. Jc and Ic values at selected magnetic fields and temperatures are shown in Table IV. The performance of the MOCVD-YBCO tape in a magnetic field was found to be similar to that of a high-quality PLD-YBCO tape. This finding is shown in fig. 9.

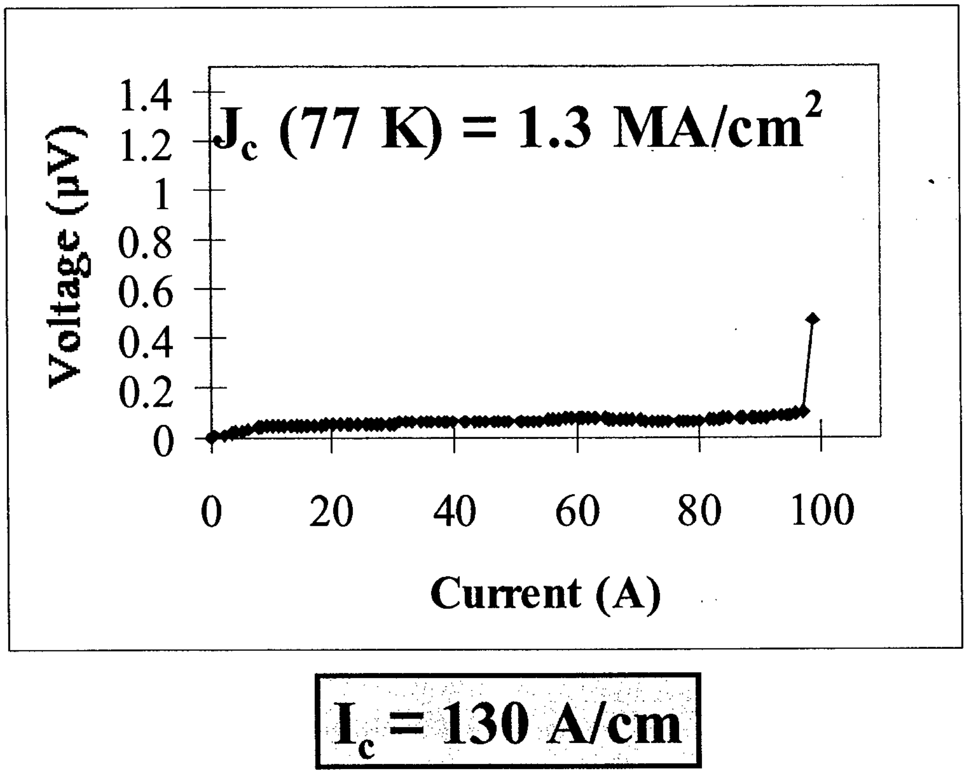


Fig. 7 I-V curve obtained from a high Jc MOCVD YBCO tape.

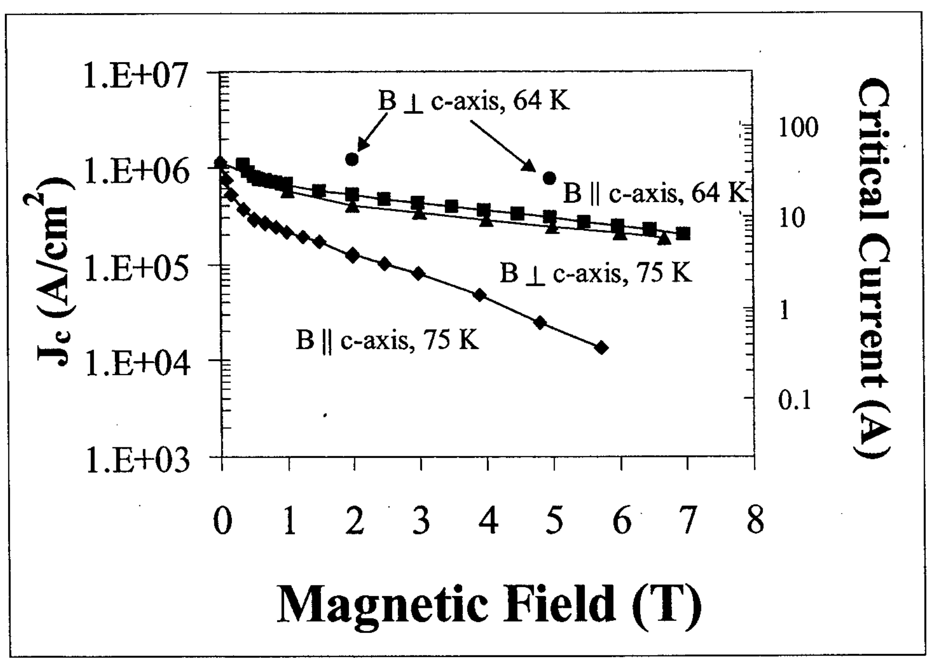


Fig. 8 Magnetic field dependence of high current YBCO tape

Table IV Performance of MOCVD YBCO tape at selected magnetic fields & temperatures

B || c (T)	J_c(kA/cm^2)	A/cm
1T, 75 K	200	20
0.3T, 64 K	1,100	109
1T, 64 K	610	66
5T, 64 K	300	31

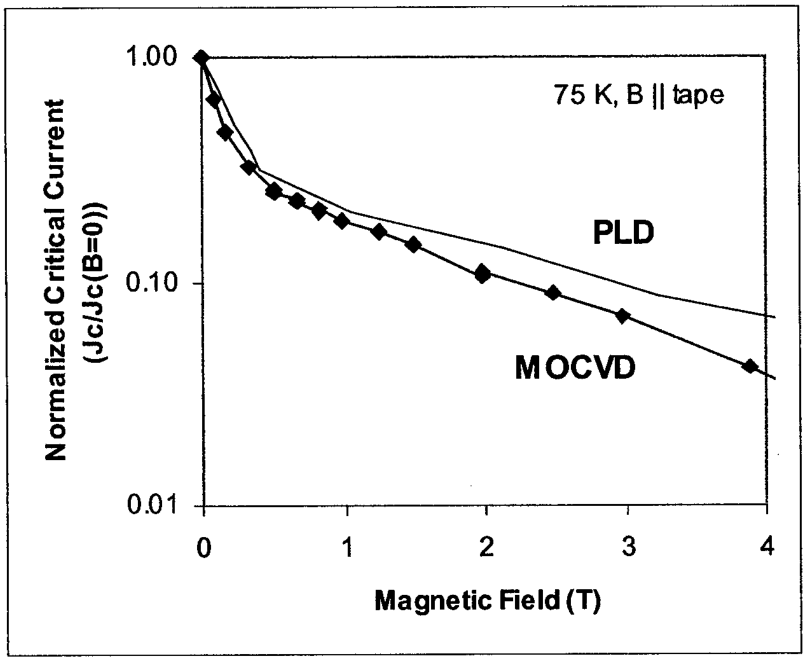
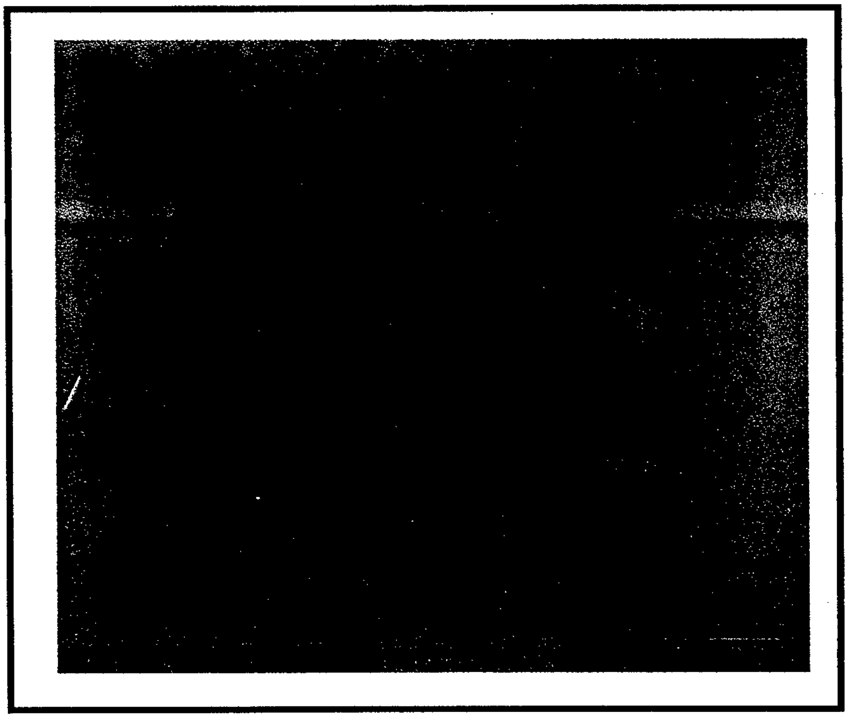


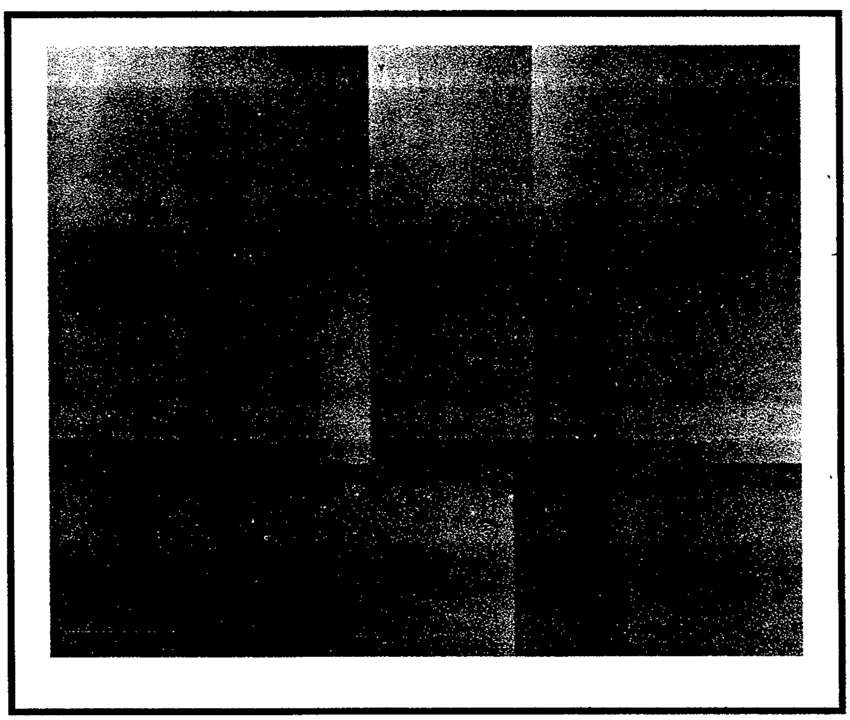
Fig. 9 Comparison of magnetic field dependence of Jc of MOCVD & PLD tapes

MOI of MOCVD YBCO :

Magneto-optic imaging of MOCVD YBCO tapes with high and low Jc was conducted at U.Wisconsin and the results are shown in fig. 10. The MOI of the high Jc tape shows a typical roof-top pattern whereas that of the low Jc tape shows more uniform flux penetration.



ZFC T=12K **H=400 Oe**



ZFC T=12K **H=40 Oe**

Fig. 10 Comparison of MOI images of high and low Jc MOCVD YBCO

Study on increasing deposition rate :

After the demonstration of high Jc on metal substrates, work in FY'02 was focussed on 2 topics, namely increasing deposition rate without reduction in Jc and demonstration of continuously moving process for MOCVD.

Two methods were used to increase the deposition rate, namely increase of the precursor flow rate and increase of precursor molarity. The effect of increase in precursor flow rate on the deposition rate is shown in fig.11. Clearly within the range studied, an one-to-one correspondence in the precursor flow rate and deposition rate was found. With a 4-fold increase in the precursor flow rate, the deposition rate also increased 4-fold indicating that all the additional precursors are being used for film growth i.e. the deposition rate is not limited by film growth kinetics in this range of deposition rate. The effect of increase in precursor molarity on the deposition rate is shown in fig. 12. Again the deposition rate increased with the molarity, but, not quite doubled with doubling of molarity.

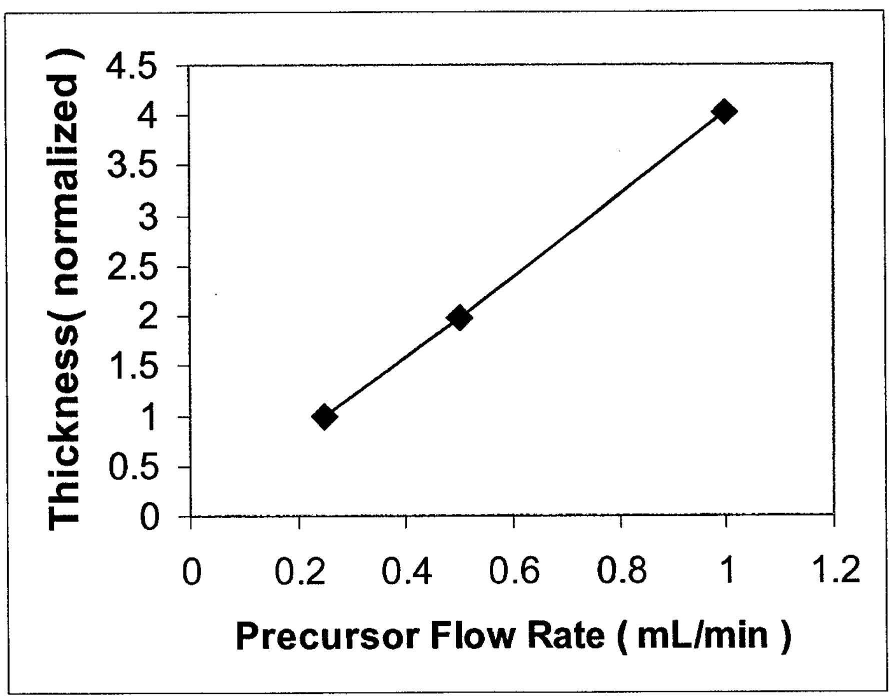


Fig. 11 Effect of increasing precursor flow rate on deposition rate of MOCVD YBCO tapes

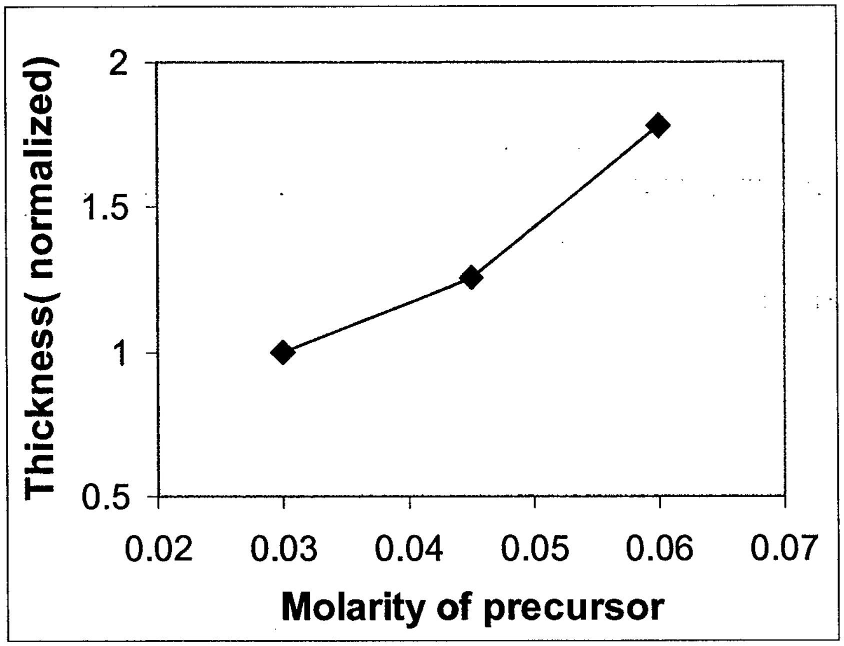


Fig. 12 Effect of increasing precursor molarity on deposition rate of MOCVD YBCO tapes

Obviously, any technique used to increase the deposition rate should not result in decreased Jc. Unfortunately, initial experiments that led to increased deposition rate led to loss of superconductivity. This is shown in fig. 13 where a 2x increase in deposition rate led to reduction of Jc from 2.8 MA/cm^2 to zero. However, after modifying the process parameters, the Jc was recovered, as shown in the figure, to a level of 2.6 MA/cm^2.

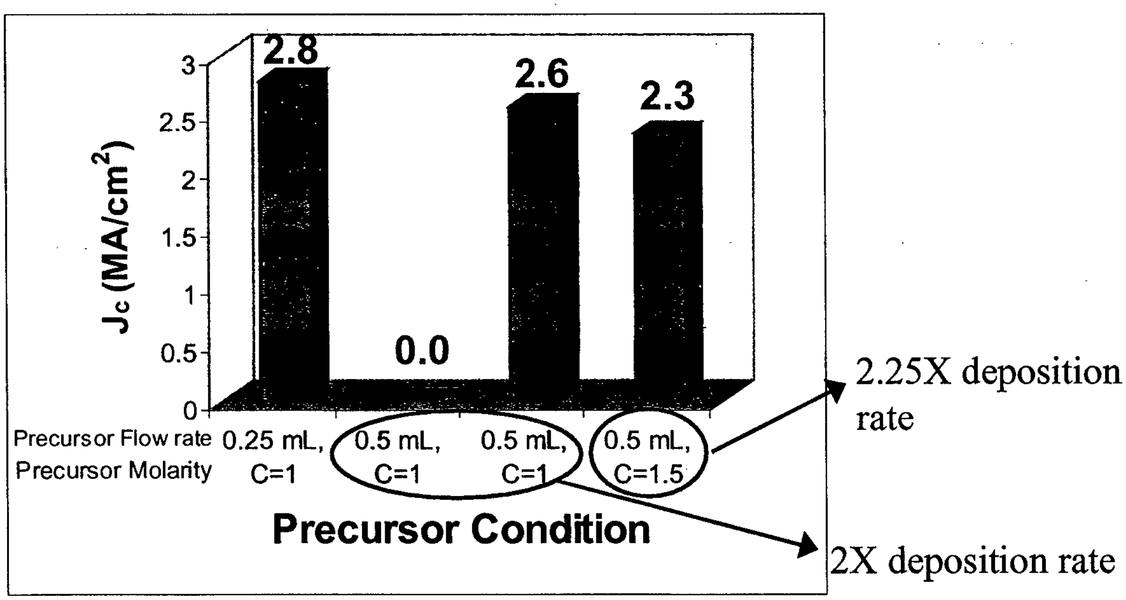


Fig. 13 Effect of increasing deposition rate on Jc of MOCVD YBCO tapes

Continuous deposition by MOCVD :

The second part of the effort in FY'02 was to demonstrate a continuous deposition process with MOCVD. For this purpose, a spooling system was set up in the same MOCVD facility. Next, a longer heater, whose hot zone is 15x that of the previously used heater was installed. These modifications are shown schematically in fig. 14 and in form a photograph in fig. 15.

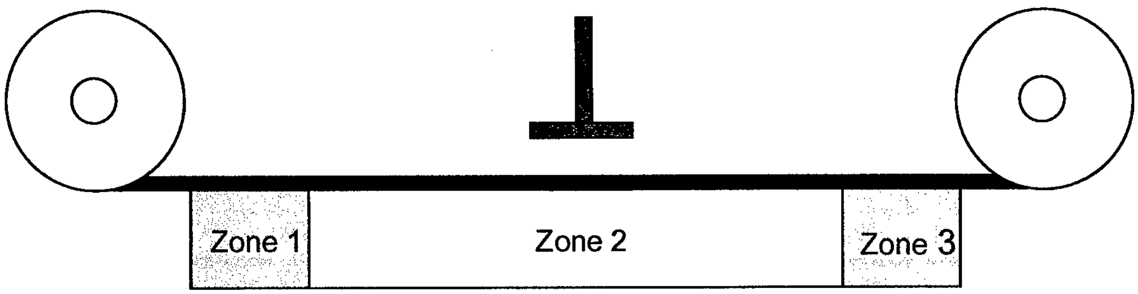


Fig. 14. Schematic of the mechanism used for continuous MOCVD on moving tape.

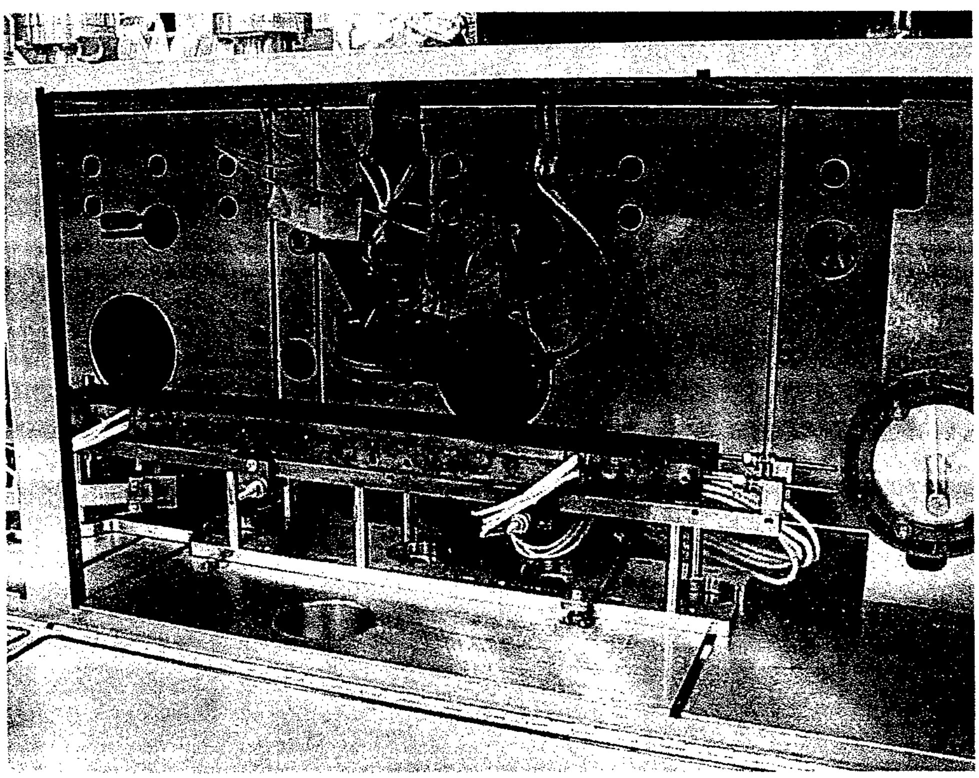


Fig. 15. Photograph of the mechanism used for continuous MOCVD on moving tape.

Figure 16 shows a photograph of a tape being processed over the longer heater. As shown in figures 14 and 15, the showerhead was not modified and therefore the deposition zone was limited, and was substantially less than the hot zone. In order to determine the length of the deposition zone, YBCO films were deposited on stationary substrates which were held under tension by the spooling mechanism.

Fig. 16 Photograph of a MOCVD tape processed continuously over the long heater

Figure 17 shows the variation in thickness, Ic, and Jc of a YBCO tape deposited over 10 cm using the above approach.

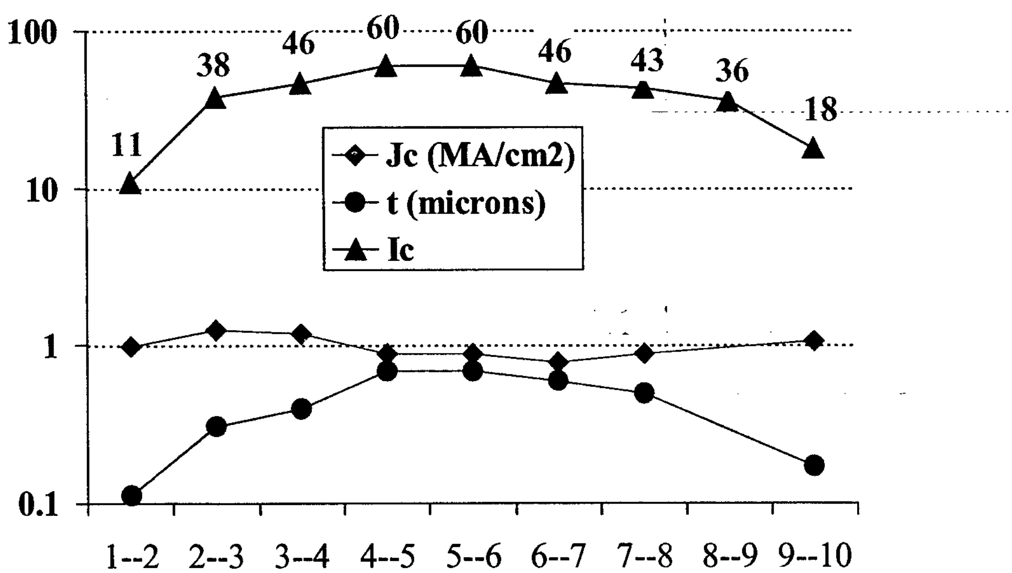


Fig. 17 Distribution of thickness, Ic, and Jc over a 10 cm long tape fabricated by MOCVD in a stationary mode.

It can be seen from the figure that the thickness varied from 0.1 to 0.8 microns because of the limited size of the showerhead. This indicates that the deposition zone is about 10 cm in length. Because of the varying thickness, the Ic varied from 11 to 60 A as shown in fig. 17. However, Jc was uniform at approximately 1 MA/cm^2 over the entire length.

Next, the film thickness was increased in order to increase the Ic, again in stationary deposition. Figure 18 shows the result from this experiment. As shown in the figure, the thickness at the end of the deposition zone increased to about 0.8 micron and in the middle to about 1.1 micron. While this led to a higher current at the ends, the current in the middle of the tape was reduced. Moreover, the Jc as a whole was suppressed, and even more in the middle.

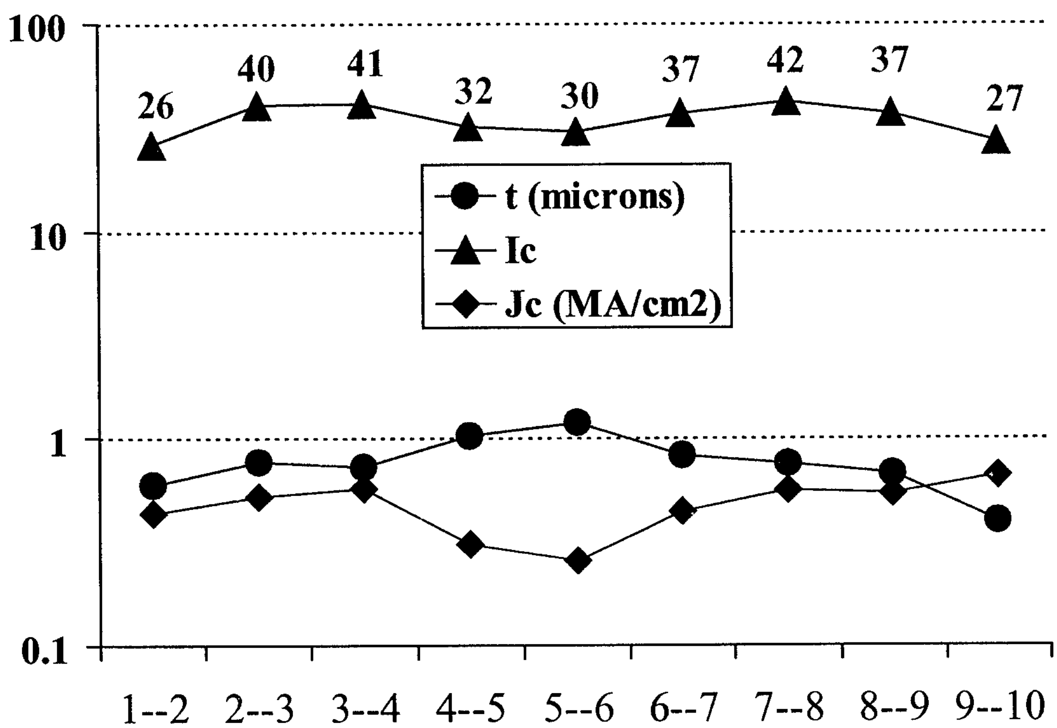


Fig. 18 Distribution of thickness, Ic, and Jc over a 10 cm long tape fabricated by MOCVD in a stationary mode with thicker YBCO.

In order to understand the reason for the suppressed Jc in the middle of the tape, as shown in fig. 18, microstructural analysis was conducted in the mid section of the tapes described in figs 17 and 18. The results are shown in figs 19 and 20 respectively. While

fig. 19 shows a typical microstructure of a high Jc MOCVD tape, fig._20 exhibits substantial a-axis grains, which could have resulted in lowered Jc. A-axis grains were found to be one of the 5 microstructural causes for reduced Jc in thicker films as described in the FY'02 report for our contract # F49620-00C-0021

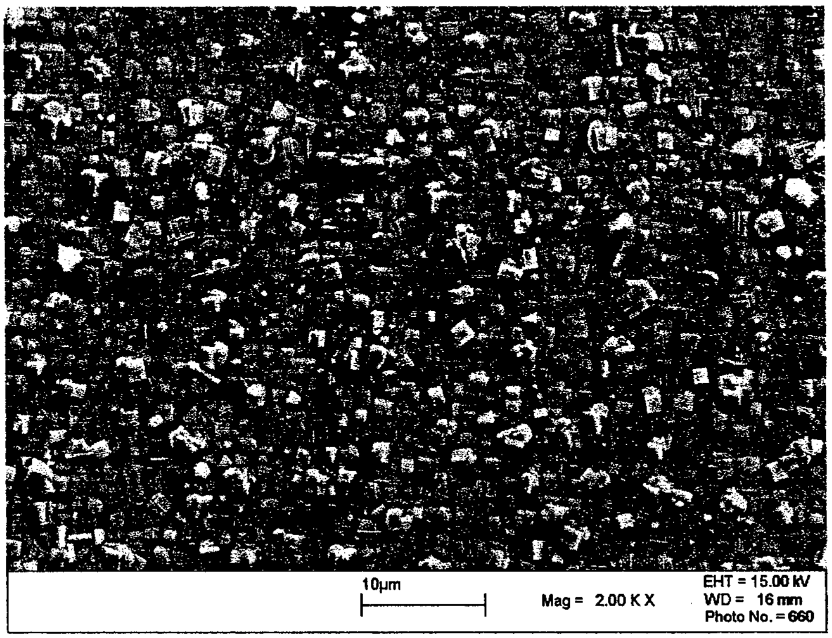


Fig. 19 Microstructure of the center part of the YBCO tape described in fig. 17

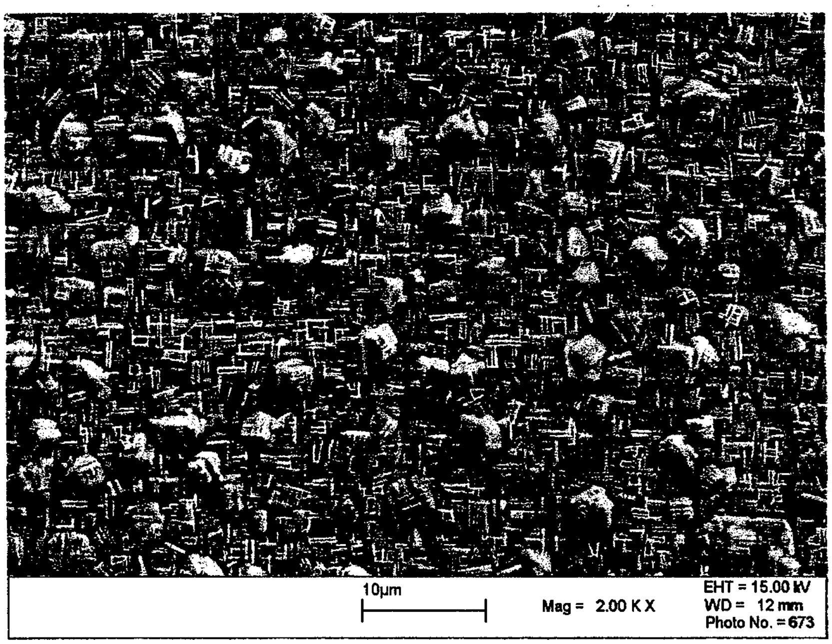


Fig. 20 Microstructure of the center part of the YBCO tape described in fig. 18

Next, YBCO deposition was begun on continuously moving tapes, up to 10 cm in length. The performance of these tapes was found to be closely correlated to the texture of the IBAD buffer layer. This finding in shown in Table V. With our best textured IBAD tape, the highest current of 135 A was achieved over 6 cm of continuously moving MOCVD tape. The best section of this tape showed a Ic of 150 A. A current-voltage characteristic (I-V curve) obtained from this section is shown in fig. 21.

Table V. Jc obtained over 6-10 cm YBCO tapes fabricated by continuous MOCVD

IBAD in-plane texture	End-to-end Ic 10 cm (A)	Best Ic section (A)
19°	60	85
16°	85	105
11°	135 (6 cm)	150

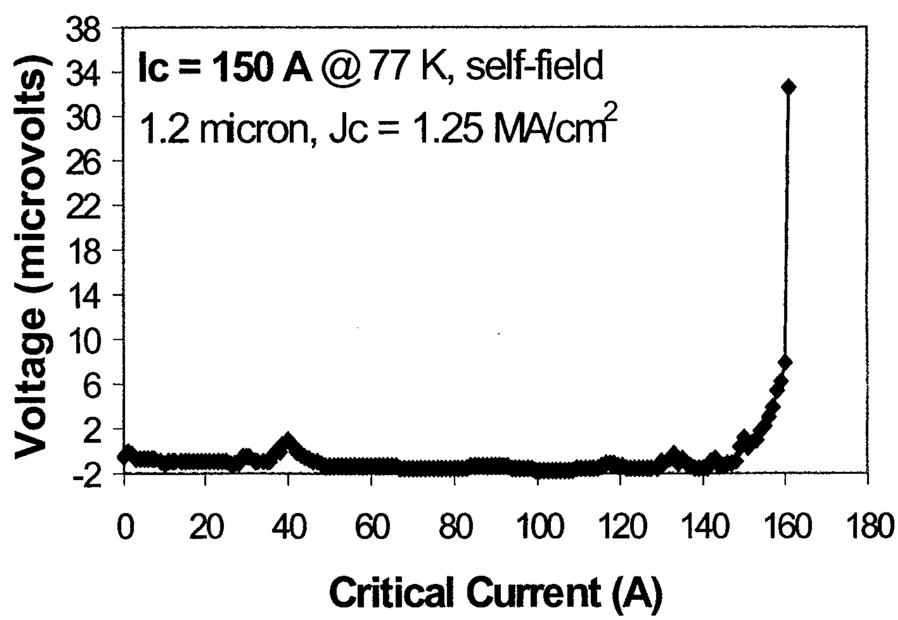


Fig. 21 I-V curve obtained from a section of a continuously processed MOCVD tape

Finally, the MOCVD process was found to be very reproducible. 4 MOCVD runs were done under identical conditions with different IBAD tapes (of same texture). Results from these runs are summarized in fig. 22. As shown in the figure, the performance was found to be uniform over the length of each tape and reproducible from run to run.

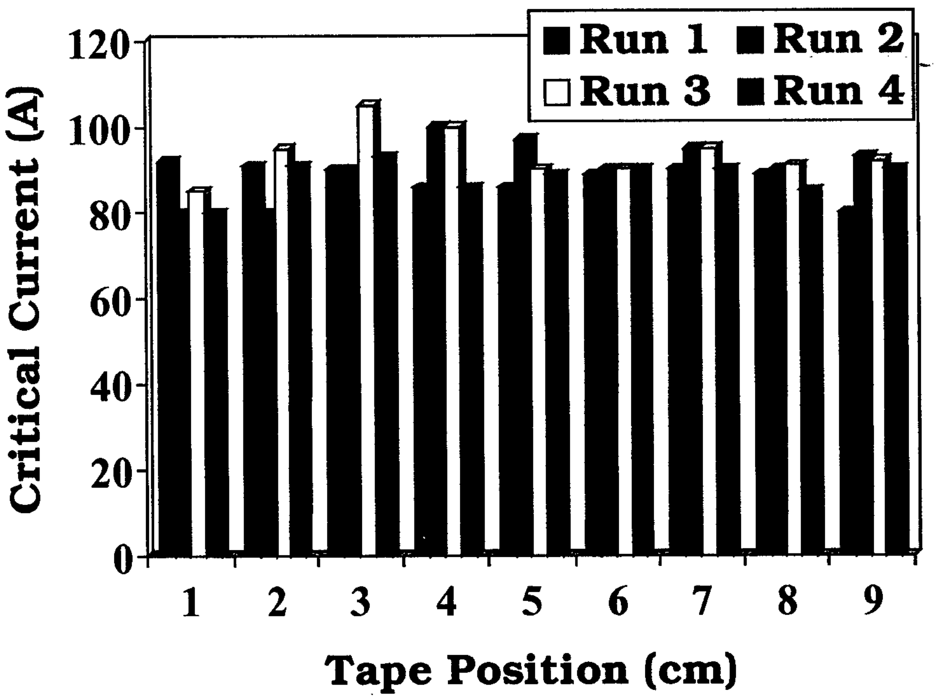


Fig. 22 Distribution of Ic over 10 long continuously processed MOCVD tapes over 4 runs conducted under identical conditions

In summary, over a five-year course of this program, we have demonstrated that the MOCVD process is a very viable technique to produce high current coated conductor. MOCVD process parameters were optimized, relationships between process, microstructure and performance understood, and several advancements were made in MOCVD hardware specifically for fabrication of YBCO coated conductors.

Personnel Supported :

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Dr. Jodi Reeves, Sr. Materials Scientist (FY'02, FY'01)

Dr. Venkat Selvamanickam, Program Manager (FY'01,'00,'99,'98)

Dr. Nghia Vo, Characterization Engineer (FY'01)

Mr. Gene Carota, Process Engineer (FY'00)

Mr. George Galinsksi, Process Engineer (FY'99, FY'98)

Publications :

1. V. Selvamanickam et al, "Scale up of High-Performance Y-Ba-Cu-O Coated Conductors", submitted for Proc. Applied Supercond. Conference. Houston, August 2002.
2. V. Selvamanickam et al, "High-Current Y-Ba-Cu-O Coated Conductor using Metal Organic Chemical-Vapor Deposition and Ion-Beam-Assisted Deposition", *IEEE Trans. Appl. Supercond.* 11, 3379, 2001
3. V. Selvamanickam et al., High-current Y-Ba-Cu-O superconducting films by metal organic chemical vapor deposition on flexible metal substrates, Physica C: 333(3-4) (2000) pp. 155-162
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7. V. Selvamanickam, A. Ivanova, D. B. Fenner, T. Thurston, M. S. Walker, A. E. Kaloyeros, and P. Haldar, "Fabrication of Biaxially-textured thick film Y-Ba-Cu-O Superconductor by MOCVD on cube-textured metal substrates", *High Temperature Superconductors : Synthesis, Processing, and Large-Scale Applications II*, ed. U. Balachandran and P. J. McGinn, p.165, TMS Publication, Warrendale, PA, 1997.

Interactions/Transitions :

a. Conference Presentations (in reverse chronological order) :

1. Applied Superconductivity Conference, Houston, August 2002
2. MRS Workshop on Coated Conductors, Gatlinburg, July 2002
3. DOE Annual Review, Washington D.C. July 2002
4. AcerS Annual Meeting, St. Louis, April 2002
5. TMS Annual Meeting, Seattle, February 2002
6. MRS Fall Meeting, Boston, November 2001
7. ISTEC-MRS workshop, Honolulu, June 2001
8. DOE Annual Review, Washington D.C. August 2001.
9. MRS Fall Meeting, Boston, November 2000
10. Appl. Supercond. Conf. Virginia Beach, Sep. 2000.
11. DOE Annual Peer Review, Washington, D.C., July 2000.
12. DOE Wire Workshop, FL, February, 2000.
13. MRS Fall Meeting, Boston 1999
14. DOE Wire Workshop, Cocoa Beach, FL, January 1999.
15. MRS Fall Meeting, Boston 1998
16. Applied Superconductivity Conference, Palm Desert, 1998.
17. MRS Fall Meeting, Boston 1997

b. Interaction :

IGC-SuperPower has been collaborating WPAFB in the development of YBCO coated conductors. Samples of MOCVD tapes have been provided to AFRL for XPS analysis. MOI of MOCVD tapes has been conducted with U. Wisconsin. IGC-SuperPower has augmented the AFOSR-funded program through CRADAs with

Argonne National Lab (ANL) and Los Alamos National Lab (LANL). ANL and LANL have been providing IBAD-buffered metal substrates for YBCO deposition at Intermagnetics. Current density measurements of Intermagnetics' samples have been conducted at LANL to verify the J_c values as well as obtain extended information. SEM and FIB analysis have been conducted by X-ray Diffraction by IGC staff and students supported by IGC-SuperPower at and U. Albany and Rensselaer Polytechnic Institute. IGC-SuperPower has also been working with WPAFB's contract personnel from UES through SBIR programs. IGC-SuperPower has been supporting small businesses such as UES and Epion to conduct research on YBCO coated conductors through joint SBIR-type research. These companies have assisted Intermagnetics in surface roughness characterization by Atomic Force Microscopy (AFM) as well deposit additional buffer layers by r.f. sputtering and PLD.

c. Transition :

The ongoing AFOSR program will have a large impact on ongoing materials and device development programs at Intermagnetics. *The AFOSR program has been a critical program at IGC-SuperPower for the development of YBCO coated conductors.* The success of the program will lead to a high-performance and potentially lower cost replacement for Bi-2223 conductor. Bi-2223 conductor is currently the main HTS conductor available in long lengths for all device programs at Intermagnetics such as transformers, cables, fault-current controllers, and generators. Based on its superior performance and potential lower cost, YBCO is the clear choice for HTS conductor for all these devices. *IGC-SuperPower recognizes this fact and is strongly supporting the AFOSR program through funds for capital equipment including the MOCVD facility and IBAD facility.* IGC-SuperPower has spent more than $ 250 k of its funds towards capital equipment for MOCVD facility. This is in addition to the purchases of new characterization equipment such as Energy Dispersive X-ray Spectroscopy, X-ray Diffractometer, Field Emission SEM, and Optical Profilometer, all of which are used for this program. The AFOSR program will eventually enable the fabrication of a high

performance superconducting tape that can find wide use in military, electric power, magnetic, medical and applications.

8. Inventions :

A Provisional Patent on "Fabrication of High Current Coated High Temperature Superconducting Tapes" filed on Aug. 7, 2000 which was converted to a full utility patent filed on Aug. 3, 2001 (PCT WO 02/056420 A2).

9. Honors & Awards :

None

10. Research Planned for Following Year :

This is the end of the 5-year program. A parallel program is underway at IGC-SuperPower, in which the effect of increasing film thickness on the Jc of MOCVD films is being examined. In the next year of that program (which ends in March '03), the work will be focussed on developing process solutions to solve the problem of decrease in Jc in thick films. Details have been provided in a separate proposal submitted earlier. As outlined in that proposal, our main objective is to achieve high Jc in thick films (> 500 A/cm at self-field).

9 different process solutions were proposed to address the 5 different microstructural causes that has been found to be responsible for lowered Jc in thick YBCO films. All the solutions address the fundamental materials science of YBCO film growth on metal substrates. We will continue to use various tools to understand the microstructure of the thick YBCO films - such as FESEM, EDS, FIB, XRD, profilometry, Auger Spectroscopy, AFM, TEM, XPS & MOI.